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Date of mailing (day/month/year) 27 January 1999 (27.01.99)	
International application No. PCT/FI98/00509	**Applicant's or agent's file reference** 7D57PCp
International filing date (day/month/year) 12 June 1998 (12.06.98)	**Priority date** (day/month/year) 13 June 1997 (13.06.97)
Applicant LEINO, Hannu, Juhani et al	

1. The designated Office is hereby notified of its election made:

[X] in the demand filed with the International Preliminary Examining Authority on:

29 December 1998 (29.12.98)

[ ] in a notice effecting later election filed with the International Bureau on:

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Telephone No.: (41-22) 338.83.38

INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 98/00509

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: D21H 17/64
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: D21H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPODOC, WPI

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	5th International Conference on New Available Technique, The World Pulp and Paper Week, Östberg, G.M.K. et al: "Use of Carbon Dioxide in the Production of Sulfate (Kraft) Pulp", June 4-7, 1996, Stockholm pp. 508-515; see page 512, line 24 - page 513, line 5; page 515, line 1 - line 8	1-13
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X✓	GB 815247 A (COLUMBIA CELLULOSE COMPANY, LIMITED), 24 June 1959 (24.06.59), page 2, line 16 - line 41	1-13
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X	US 5378322 A (DERED HORNSEY), 3 January 1995 (03.01.95), claims	1-13
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[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
6 October 1998	12.10.1998
Name and mailing address of the ISA: Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Anneli Jönsson Telephone No. +46 8 782 25 00

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 98/00509

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0281273 A1 (THE GROUP, INC.), 7 Sept 1988 (07.09.88), column 2, line 6 - line 19; claims -- --------	1-13

INTERNATIONAL SEARCH REPORT

Information on patent family members

27/07/98

International application No.
PCT/FI 98/00509

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB 815247 A	24/06/59	NONE	
US 5378322 A	03/01/95	CA 2069713 A	28/11/93
		DE 69315119 D,T	05/03/98
		EP 0572304 A,B	01/12/93
		SE 0572304 T3	
		ES 2108839 T	01/01/98
		FI 932435 A	28/11/93
		JP 6299496 A	25/10/94
EP 0281273 A1	07/09/88	SE 0281273 T3	
		AU 1175188 A	01/09/88
		DE 3882395 T	02/12/93
		FI 880918 A	28/08/88
		JP 1045887 A	20/02/89
		JP 2582838 B	19/02/97

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International application No.
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		FI 932435 A	28/11/93
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EP 0281273 A1	07/09/88	SE 0281273 T3	
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		DE 3882395 T	02/12/93
		FI 880918 A	28/08/88
		JP 1045887 A	20/02/89
		JP 2582838 B	19/02/97

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 88 30 1311

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
X	US-A-2 114 809 (F.G.RAWLING) * Figure; page 1, left-hand column, line 33 - page 2, left-hand column, line 46; page 3, right-hand column, lines 16-33; page 4, left-hand column, lines 15-23 *	1,3,7, 12,14	D 21 H 3/74 D 21 D 3/00
X	US-A-1 993 265 (C.P.DYER) * Page 1, right-hand column, line 21 - page 2, left-hand column, line 74 *	1,7,11, 12,14	
A	DE-C- 235 821 (W.BRÄUNER et al.) * Whole document * & FR-A-435 013	1,11	
A	US-A-3 112 242 (H.L.JONES)		

TECHNICAL FIELDS SEARCHED (Int. Cl.4)

D 21 D
D 21 H

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	02-06-1988	NESTBY K.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
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(74) Agent: BORENIUS & CO. OY AB; Kansakoulukuja 3, FIN–00100 Helsinki (FI).

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(54) Title: A PROCESS FOR STABILIZING THE pH OF A PULP SUSPENSION AND FOR PRODUCING PAPER FROM THE STABILIZED PULP

(57) Abstract

The invention relates to a process for stabilizing the pH of a pulp suspension with buffering agents and to a process for producing paper from a stabilized pulp suspension. The alkalinity of the pulp suspension is increased by a combination of an alkali metal hydroxide feed and a carbon dioxide feed. Said feeds provide a significant buffering effect which stablilizes the pH of said pulp suspension for the paper making process.

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A process for stabilizing the pH of a pulp suspension and for producing paper from the stabilized pulp

The invention relates to a process for stabilizing the pH of a paper making pulp suspension with buffering agents and to a process for producing paper from such a stabilized pulp suspension.

During the last ten to fifteen years many paper makers have converted their processes from acidic to neutral pH for a number of reasons, e.g. to gain increased strength and to be able to use calcium carbonate, $CaCO_3$, as a filler. The expression "neutral pH" corresponds in these processes to a pH in the short circulation of approximately 7-8,5, most preferably 7-8. This applies to paper produced from chemical, mechanical and recycled pulp, bleached or unbleached.

If the paper making pulp is acidic when entering the stock preparation and the short circulation is run at a neutral or alkaline pH, the traditional way of raising and controlling the pH is to add sodium hydroxide, NaOH. NaOH is, however, a very strong base, which means that only small amounts are needed for pH adjustments. Any over-dosage will cause a too big pH increase, which means that it is difficult to perform the pH adjustment in a controlled way. This is due to the low inherent buffering ability of a pulp suspension. The paper maker could end up in a situation with varying pH of the entering pulp, which has a negative impact on paper quality and paper machine runnability.

Through the stock preparation and the short circulation a number of paper chemicals and dilution waters are added, some of which are acidic and therefore decrease the pH of the pulp. The paper maker could therefore end up with a too low pH in the short circulation and would be once again forced to pH adjust using NaOH. The pH may also change at refining or in storage towers.

If the paper making pulp is alkaline instead when entering the stock preparation and if the short circulation is run at a neutral or alkaline pH, there is naturally no need to use any NaOH for pH control. The paper maker must however make sure that the pH is high enough to avoid ending up with too low a pH after addition of acidic paper chemicals.

In the prior art one way of avoiding ending up with too low pH values has been to add dissolved sodium bicarbonate, $NaHCO_3$, to the pulp. The $NaHCO_3$ dissociates in the pulp

suspension forming bicarbonate ions, HCO_3^-, which have a buffering effect and therefore counteract any pH decrease. $NaHCO_3$ is a solid powder, which is generally supplied in so called big-bags, and the paper mill needs space for handling, equipment for dissolving and tanks for storage. The $NaHCO_3$ is messy to work with, when in contact with moisture or water.

In the non-acidic sizing of paper with alkylketene dimers bicarbonate ions have been used to catalyze the reaction between the alkylketene dimers and the cellulose. According to US Patent 5,378,322 the bicarbonate ions may be generated by dissociation of carbon dioxide CO_2 in the aqueous pulp.

Carbon dioxide is a gas, which easily dissolves under alkaline conditions, e.g. in water or a pulp suspension forming carbonic acid and/or bicarbonate ions according to the reaction:

$$CO_2 + H_2O <=> H_2CO_3 <=> H^+ + HCO_3^-$$

At a high pH, especially greater than 10, the predominant reaction is

$$CO_2 + OH^- <=> HCO_3^-$$

In the recycling of gypsum-containing waste or broke paper the accumulation of calcium sulfate poses a problem since the solubilized calcium sulfate may precipitate and disturb the paper making process. According to US Patent 5,262,006 this problem may be overcome by adding carbonate ions and/or bicarbonate ions to the aqueous pulp suspension and by adjusting the pH to an alkaline value to precipitate the calcium as calcium carbonate. The bicarbonate ions may be created *in situ* for example by first adding a suitable soluble metal hydroxide and then adding carbon dioxide. According to the Patent, the calcium carbonate generation from calcium sulfate with carbon dioxide requires a close control of the pH since carbon dioxide has the effect of lowering the pH so that there is a risk of the pH becoming too low for the carbonation process.

Carbon dioxide has also been used for the pH control of pulp suspensions, for instance, in a process described in EP Patent 0 281 273, wherein gaseous carbon dioxide is introduced to adjust and maintain the pH at a value which is preferably between 7.0 and 5.5.

Thus, in the prior art carbon dioxide has been used to create bicarbonate ions for its catalyzing, carbonating or pH lowering effect. However, a paper maker also has the need

to obtain a stable pH so that chemical additions and various processing steps do not cause an unwanted fluctuation of the pH.

An object of the present invention is thus to provide a pH stabilization of aqueous pulp suspensions.

Another object of the invention is to provide a paper making pulp suspension having an increased alkalinity, i.e. a resistance to pH change.

A further object of the invention is to provide a technically advantageous process for buffering a pulp suspension.

An object is also to provide a pulp suspension having a controlled pH which is maintained at a desired level.

An object of the invention is also to provide a process for producing paper from a pulp suspension having a stabilized pH.

The invention according to the present application is defined in the appended claims, the contents of which are included herein by reference.

Consequently, the present invention relates to a process for stabilizing the pH of a pulp suspension, wherein the alkalinity of a paper making pulp suspension is increased by adding thereto a combination of an alkali metal hydroxide feed and a carbon dioxide feed which feeds substantially counter each other's pH changing effect, said feeds being provided in an amount sufficient to achieve a significant buffering effect of said pulp suspension for paper making.

The feeding of a hydroxide and carbon dioxide in substantially countering amounts increases the level of alkalinity forming ions in the suspension without actually affecting its pH. Increasing the alkalinity stabilizes the suspension against pH fluctuations caused by subsequent additions of acidic or basic fluids and provides a stable pH throughout the succeeding steps.

Alkalinity is a measurement of "acid resistance", i.e. the content of buffering ions in a liquid or pulp suspension, which counteracts pH decrease at addition of hydrogen ions, H^+. One way of expressing alkalinity is the amount of HCO_3^- and CO_3^{2-} ions in grams

per liter. When a combination of hydroxide and carbon dioxide is used according to the present invention, the alkalinity of the pulp suspension is raised, thus creating larger "acid resistance".

The hydroxide used according to the invention should preferably be added prior to the addition of the carbon dioxide to ensure that the carbon dioxide is added under alkaline conditions.

The "amount sufficient to achieve a significant buffering effect" should be taken as meaning an amount providing a substantial and recognizable buffering effect in the pulp suspension. The amount normally varies depending on the characteristics of the pulp and on the conditions of treatment. A person skilled in the art will be able to calculate the required amount based on his general knowledge or by simple tests made on the actual pulp suspension, as indicated, for example in Example 9 of this specification.

Typically the amount of NaOH in the buffering combination will be between about 0.5 and 5 kg NaOH/ton dry cellulose and the amount of carbon dioxide in said combination will be between about 0.5 and 5 kg CO_2/ton dry cellulose. A typical buffering combination would include about 2 to 3 kg of NaOH and CO_2 per ton of dry cellulose. It is within the scope of the present invention to use more or less than the above mentioned amounts of both components, but in general it will be uneconomical to use more than what is required for a desired buffering action.

It is clear that the excess of either NaOH or CO_2 which is additionally used for pH adjusting purposes may significantly exceed the above amounts which provide the buffering effect.

The buffering effect obtained by the addition of NaOH and CO_2 in this way corresponds to the one obtainable by an addition of dissolved $NaHCO_3$ but it has the advantage that the space consuming and messy handling of solid $NaHCO_3$ is avoided. Sodium hydroxide, on the other hand, is a chemical which is abundantly available in the paper mill since it is used for many other purposes. Carbon dioxide gas may be generated on site or may be bought as desired. Feeding of carbon dioxide into the suspension is technically clean and easy.

A further advantage resides in that the hydroxide and carbon dioxide used according to the present invention may serve the dual purpose of increasing the alkalinity and of adjusting

the pH. Thus, according to a preferred embodiment of the invention, the pH of said pulp suspension is increased by adding an excess of alkali metal hydroxide such as aqueous sodium hydroxide or decreased by adding an excess of carbon dioxide.

The carbon dioxide is preferably in gaseous form, although it may be added as an aqueous liquid by dissolving gaseous or solid carbon dioxide in water. The hydroxide and carbon dioxide may be combined prior to feeding to the pulp suspension although it is preferred to feed them directly into the pulp circulation system such as to a pipe leading to a stock preparation tank.

The pulp suspension is preferably buffered by said combination to a pH between about 7 and 9.

According to the preferred embodiment of the invention the alkalinity of the pulp suspension is increased by providing a substantially equal molar amount of alkali metal hydroxide and dissolved carbon dioxide, said amount being sufficient to provide a significant buffering effect at about pH 8.

The pulp suspension may be bleached or unbleached chemical or mechanical pulp although the preferred pulp is bleached chemical pulp.

Calcium carbonate may advantageously be used as a filler for the pulp, since the stabilized pH will ascertain that the filler remains in solid form in the suspension. A fluctuation of the pH down to 6 or 5.5, for instance due to an inflow of circulating white water at such a pH, might dissolve the carbonate filler. Such an undesirable effect will be effectively prevented by the buffering action of the present invention.

The present invention also relates to a process for producing paper, said process comprising the steps of

- providing a paper making pulp suspension;
- increasing the alkalinity of said pulp suspension by adding thereto a combination of an alkali metal hydroxide feed and a carbon dioxide feed which feeds substantially counter each others pH changing effect, said feeds being provided in an amount sufficient to achieve a substantial buffering effect of said pulp suspension for paper making;
- optionally adjusting the pH of said pulp suspension to a desired value between 7 and 9 by adding an alkaline agent such as NaOH or an acidic agent such as CO_2;
- forming said pulp suspension into a web; and

- drying said web to form paper.

The production of paper according to the present invention is performed in a conventional way in all other respects except for the increase of the alkalinity of the pulp prior to the short circulation. Such paper making processes are well known in the art and it is not considered necessary to describe them here in any greater detail.

The invention will now be illustrated with a few examples which should not be considered as limiting the invention in any way.

Example 1
(Reference example)

In a partly integrated paper mill bales of fully bleached kraft market pulp are introduced into a pulp slusher. The pH in the slusher is adjusted with aqueous NaOH to a pH of approximately 11.

After slushing, paper making chemicals and dilution water having an acidic effect are added to the slushed pulp suspension. As a consequence of this, the pH of the pulp suspension decreases from pH 11 to about pH 6.5-6.8. This pH is too low for the short circulation, which is run at a pH level of 7-7.5. Thus, the pH is again adjusted by an addition of aqueous NaOH.

Because of the strong basic action of NaOH it is difficult to achieve an exact pH control in this way. Over-dosing leads to a too high pH.

Example 2

The process of Example 1 is changed in order to improve the situation, so that a combination of NaOH and CO_2 is added to the pulp slusher. The amount of NaOH added to the suspension is substantially increased compared to the process of Example 1. A countering amount of CO_2 is added to provide a pH of approximately pH 9.

After slushing, the same paper chemicals as in Example 1 are added to the slushed pulp suspension. Because of the buffering effect of the combined NaOH and CO_2, the acidic additions lower the pH only to pH 7.2. This is a suitable pH for the short circulation and there is no need for any pH control using NaOH.

Example 3
(Reference example)

In a paper mill a kraft pulp suspension having a pH of 5.1 is fed to a storage tower. Prior to the entry into the tower, the pH of the pulp suspension is adjusted to pH 8 by an addition of aqueous NaOH into the pipe leading to the tower. The pulp suspension is fed from the storage tower to a refiner. At refining the pH decreases to about pH 6.

The low pH causes problems in the subsequent sizing of the pulp.

Example 4

The process of Example 3 is repeated with the exception that instead of feeding only the required amount of NaOH into the pulp suspension, an excess of NaOH is fed first into the pipe followed by feeding gaseous CO_2 into the pipe counteracting the excess NaOH. The resulting suspension again has a pH of 8 when entering the storage tower.

From the storage tower the pulp is fed to the refiner. At refining the pH remains at about pH 8 and there are no problems at sizing.

Example 5

In a laboratory trial a pulp suspension having a pH of 5.1 was used. The pH of the pulp suspension was adjusted to pH 8 by using a) only NaOH and b) by using an excess of NaOH in combination with CO_2. The buffering effect of the suspension was tested with a strong acid (alum, pH 3.0).

The results are shown in Table 1 below.

Table 1

	pH^1	NaOH (ml)	CO_2 (g)	pH^2	alum (ml)	pH^3
a) (NaOH)	5.1	2	-	8.0	1	6.4
b) (NaOH + CO_2)	5.1	3	1	8.0	1	7.9

[1] the pH of the initial pulp suspension
[2] the pH after addition of NaOH / NaOH + CO_2
[3] the pH after addition of acid

In the above process 1 ml of the aqueous NaOH corresponds to about 2.5 kg/ton cellulose calculated on the dry weight basis; 1 g of CO_2 similarly corresponds to about 2.5 kg/ton cellulose; and 1 ml of alum corresponds to about 2.8 kg/ton cellulose.

The above results clearly show that the addition of countering amounts of NaOH and CO_2 stabilize the pH of the pulp suspension.

Example 6
(Reference example)

A pulp suspension was stored in a pulp storage tank in a pulp mill at a pH of 5.5 to 6. The pulp was fed at this pH at a consistency of 3 to 4% to the paper mill via a press which increased the consistency to about 10%. The pulp was fed at this consistency to the paper mill storage tank at a pH of 5.5 to 6.

White water from the paper machine was fed to the storage tank at a pH of 7.5 to 8. The paper was produced with $CaCO_3$ as filler. Some of the filler circulated with the white water to the storage tank. When entering the storage tank the $CaCO_3$ in the white water dissolved as it met the pulp suspension at pH 5.5 to 6.

The dissolving of the $CaCO_3$ caused loss of filler and in addition thereto the calcium ions increased the hardness of the water and caused precipitations at unwanted positions.

From the storage tank the suspension was fed to a refiner. For improving the refining the pH of the pulp suspension was adjusted to 7.5 with NaOH before the refiner. Careful control of the NaOH feed was required to avoid dosing too much or too little.

In the refiner the pH decreased to 6 to 6.5 which meant increased energy consumption at refining. The pH was too low for the sizing. Thus, the pH had to be adjusted again for the sizing.

Example 7
(Reference example)

The process of Example 6 was repeated with the exception that aqueous $NaHCO_3$ was fed to the paper mill storage tank to obtain the desired pH of about 7.5 to 8. The $CaCO_3$ in the white water was not dissolved and the problem with filler loss was resolved.

The pH of the feed to the refiner was about 7.5 and no NaOH was fed to the suspension. However, in the refiner the pH decreased as before and a pH adjustment with NaOH was required for sizing.

Example 8

The process of Example 6 was repeated with the exception that NaOH and CO_2 were fed to the pipe leading to the paper mill storage tank. The pH was adjusted to 8 and no loss of filler occurred in the storage tank.

The pH of the feed to the refiner was about 8 and no NaOH was fed to the suspension. At refining the alkalinity of the suspension counteracted the pH lowering effect in a sufficient degree to retain the pH at 7.5 to 8.

There was no need for a pH adjustment for sizing. The pulp was used for the production of paper with an excellent result.

Example 9

A pulp suspension having a 10% consistency and a pH of 7.4 was divided into two lots, Pulp 1 and Pulp 2, respectively, weighing 2030 g each. The pH of the pulp suspension was adjusted on one hand with sulfuric acid (a 10% by weight aqueous solution of H_2SO_4) and on the other hand with gaseous carbon dioxide and a combination of 1 M sodium hydroxide and carbon dioxide gas.

The pH of Pulp 1 was adjusted to 6.2 with said sulfuric acid. The pH of Pulp 2 was adjusted to 6.2 by adding 15 ml sodium hydroxide and 1,02 g of carbon dioxide. The resulting pulp suspensions were titrated with 10% sulfuric acid to study their respective resistance to pH change.

The results are indicated in Table 2 below.

Table 2

Titration H_2SO_4 (kg/t)	pH Pulp 1	pH Pulp 2
0	6.22	6.20
1.4	6.18	6.20
2.4	6.10	6.21
4.7	6.03	6.17
10.6	5.82	6.12

The sulfuric acid in Table 2 is indicatied in kg of 100% H_2SO_4 per ton of dry cellulose.

For the above adjustment of the pH and alkalinity of Pulp 2, NaOH was added in an amount of 2.9 kg/ton cellulose and CO_2 was used in an amount of 5.1 kg/ton cellulose. The CO_2 was used both for pH adjustment and to counter the addition of sodium hydroxide.

The test shows that fairly small amounts of a combination of sodium hydroxide and carbon dioxide provides an effective buffering action. The above final pH of 6.12 would render Pulp 2 fully useful for papermaking purposes, while the final pH of Pulp 1 would be too low.

It is evident that the invention may be varied in a great number of ways which are obvious to those skilled in the art without deviating from the scope of the claims.

Claims

1. A process for stabilizing the pH of a pulp suspension, c h a r a c t e r i z e d in that the alkalinity of a paper making pulp suspension is increased by adding thereto a combination of an alkali metal hydroxide feed and a carbon dioxide feed which feeds substantially counter each other's pH changing effect, said feeds being provided in an amount sufficient to achieve a significant buffering effect of said pulp suspension for paper making.

2. Process according to claim 1, c h a r a c t e r i z e d in that the pH of said pulp suspension is adjusted by adding an alkaline agent such as an excess of said alkali metal hydroxide or by adding an acidic agent such as an excess of said carbon dioxide.

3. Process according to claim 1, c h a r a c t e r i z e d in that said alkali metal hydroxide is aqueous sodium hydroxide and said carbon dioxide is gaseous carbon dioxide.

4. Process according to claim 1, c h a r a c t e r i z e d in that said alkali metal hydroxide is fed to said pulp suspension prior to the feeding of said carbon dioxide.

5. Process according to claim 1, c h a r a c t e r i z e d in that said pulp suspension is buffered by said combination to a pH between about 7 and 9.

6. Process according to claim 5, c h a r a c t e r i z e d in that the alkalinity of said pulp suspension is increased by providing a substantially equal molar amount of alkali metal hydroxide and dissolved carbon dioxide, said amount being sufficient to provide a significant buffering effect at about pH 8.

7. Process according to claim 1, c h a r a c t e r i z e d in that the pH of said pulp suspension is adjusted to a value lower than pH 8 by adding an excess of carbon dioxide.

8. Process according to claim 1, c h a r a c t e r i z e d in that the pH of said pulp suspension is adjusted to a value higher than pH 8 by adding an excess of alkali metal hydroxide.

9. Process according to claim 1, c h a r a c t e r i z e d in that said pulp suspension is chemical or mechanical pulp.

10. Process according to claim 9, c h a r a c t e r i z e d in that said pulp suspension is

bleached chemical pulp.

11. Process according to claim 1, characterized in that said pulp suspension contains and/or is intended to contain calcium carbonate filler.

12. Process according to claim 1, characterized in that said alkali metal hydroxide and carbon dioxide feeds are added to said pulp suspension flowing in a pipe leading to a stock preparation tank.

13. A process for producing paper comprising
- providing a paper making pulp suspension;
- increasing the alkalinity of said pulp suspension by adding thereto a combination of an alkali metal hydroxide feed and a carbon dioxide feed which feeds substantially counter each others pH changing effect, said feeds being provided in an amount sufficient to achieve a substantial buffering effect of said pulp suspension for paper making;
- optionally adjusting the pH of said pulp suspension to a desired value between 7 and 9 by adding an alkaline agent or an acidic agent;
- forming said pulp suspension into a web; and
- drying said web to form paper.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 98/00509

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: D21H 17/64
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: D21H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPODOC, WPI

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	5th International Conference on New Available Technique, The World Pulp and Paper Week, Östberg, G.M.K. et al: "Use of Carbon Dioxide in the Production of Sulfate (Kraft) Pulp", June 4-7, 1996, Stockholm pp. 508-515; see page 512, line 24 - page 513, line 5; page 515, line 1 - line 8	1-13
X	GB 815247 A (COLUMBIA CELLULOSE COMPANY, LIMITED), 24 June 1959 (24.06.59), page 2, line 16 - line 41	1-13
X	US 5378322 A (DERED HORNSEY), 3 January 1995 (03.01.95), claims	1-13

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" erlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
6 October 1998	12.10.1998
Name and mailing address of the ISA: Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Anneli Jönsson Telephone No. +46 8 782 25 00

INTERNATIONAL SEARCH REPORT

International application No.
PCT/FI 98/00509

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0281273 A1 (THE GROUP, INC.), 7 Sept 1988 (07.09.88), column 2, line 6 - line 19; claims -- --------	1-13

INTERNATIONAL SEARCH REPORT

Information on patent family members

27/07/98

International application No.
PCT/FI 98/00509

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
GB 815247 A	24/06/59	NONE	
US 5378322 A	03/01/95	CA 2069713 A	28/11/93
		DE 69315119 D,T	05/03/98
		EP 0572304 A,B	01/12/93
		SE 0572304 T3	
		ES 2108839 T	01/01/98
		FI 932435 A	28/11/93
		JP 6299496 A	25/10/94
EP 0281273 A1	07/09/88	SE 0281273 T3	
		AU 1175188 A	01/09/88
		DE 3882395 T	02/12/93
		FI 880918 A	28/08/88
		JP 1045887 A	20/02/89
		JP 2582838 B	19/02/97

E.Pe

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

12

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
7D57PCp		
International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/FI98/00509	12/06/1998	13/06/1997
International Patent Classification (IPC) or national classification and IPC D21H17/64		
Applicant AGA AKTIEBOLAG et al		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 6 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 2 sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   - VI ☐ Certain documents cited
   - VII ☒ Certain defects in the international application
   - VIII ☒ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
29/12/1998	12. 10. 99
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Karlsson, B Telephone No. +49 89 2399 8424

Form PCT/IPEA/409 (cover sheet) (January 1994)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/FI98/00509

I. Basis of the report

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

Description, pages:

1-10	as originally filed				

Claims, No.:

13	as originally filed				
1-12	as received on	13/08/1999	with letter of	02/08/1999	

2. The amendments have resulted in the cancellation of:

☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-12
	No:	Claims	
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1-12
Industrial applicability (IA)	Yes:	Claims	1-12
	No:	Claims	

2. Citations and explanations

see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

V

1.1 The present claim 1 defines a process for stabilizing the pH of a pulp suspension in the stock preparation of a paper machine by buffering said suspension by adding a combination of an alkali metal hydroxide and a carbon dioxide.

1.2 D3:EP-A-0 281 273, also cited in the description, discloses a process for manufacturing paper from a cellulosic pulp, which tries to improve the pH-adjustment of the alkaline pulp prior to forming paper therefrom (see D3, column 2, 1st paragraph). According to D3, gaseous carbon dioxide is added to the pulp suspension in order to adjust and maintain the pH at desired level. It is even explicitly disclosed in D3 that due to the buffering capacity of carbon dioxide, the pulp pH variations can substantially be avoided, which leads to improved properties of the resulting paper (see column 6, 1st paragraph). Moreover, it must also be considered to be a well known fact to the skilled engineer, regardless if he works in the pulp mill or paper factory, that CO_2 has a buffering capacity in aqueous solutions, i.e. in pulp suspensions (see D2: 5th Inter. conference on new available tech., The world pulp and paper week, Östberg G.M.K. (see search report), page 509, third paragraph).
Therefore, as already explained above, the skilled engineer who tries to solve the problem of adjusting the pH of pulp suspensions in the stock preparation, would surely also consider how said problem has been solved in a technically very closely related field, i.e. in the pulp mill. Thus, a skilled person would surely also consider D1:GB-A-815 247 although this document more generally deals with the manufacture of pulp without explicitly mentioning anything about the manufacture of paper therefrom. The skilled man would from this document get an indication on how the pulp suspension advantageously can be buffered, namely by using a combination of sodium hydroxide and carbon dioxide (see D1 page 2, lines 16 to 40). Thus, the combination of D3 and D1 would lead the skilled person to the subject-matter of the present claim 1. Accordingly, the requirements of Article 33.3 PCT does not appear to be satisfied.

1.3 The features of the present claims 2,3 and 4 are already known from D1 (see above reference).
The additional features of claims 6 and 7 are known from D3 (see D3, column 1; claim 1). Further, no inventive matter appears to be present in the subject-matters of claims 5,8, 9 and 10 in the light of the disclosure of D1 and D3. Thus, presently no inventive

matter appears to be present in the dependent claims 2 to 10 in the light of the disclosures of D1 and D2 (Art. 33.3 PCT).

1.4 The wording of the present claim 11 just differs from claim 1 by the fact that it refers to a "process for producing paper" instead of a "process for stabilizing the pH of a pulp suspension in a stock preparation of a paper machine". Thus, since D3 also pertains to a process for manufacturing paper, the distinguishing features of claim 11 are then, of course, known from D3 (see D3, fig.1). Hence, by the same token as explained above, the subject-matter of claim 11 does neither satisfy the requirements of Article 33.3 PCT with regard to the disclosures of D3 and D1.

1.5 The features of the present claim 12 are known from both D3 and D1 and cannot thus be considered to add any inventive matter (see above references to D3 and D1; Art.33.3 PCT).

1.6 Although the separate features of the dependent claims not appear to add any inventive matter, the clarified claim 1, amended with a specific combination of features, might still satisfy the requirements of Articles 33.2 and 33.3 PCT.

VII

2.1 D1 and D2 should be acknowledged in the description as representing closest prior art (Rule 5.1(a)(i)-(vi) PCT).

VIII

3.1 The present claim 1 refers to a method for stabilizing the pH of a pulp suspension in the stock preparation of a paper machine. The necessary process steps to accomplish said stabilizing effect have, however, not been clearly defined in claim 1 (Art.6 PCT). It is on one hand stated that the alkalinity of the pulp suspension should be increased i.e. increasing the pH, whereas, on the other hand it is stated the pH of the pulps suspension should be adjusted by adding a excess of the hydroxide component resp. carbon dioxide, i.e. the pH can be decreased or increased. Following the examples of the present specification it becomes clear that the pH of the pulp suspension can be both decreased and increased by adding a small surplus of the

resp. buffering agents.
The exact meaning of the wording " increasing the alkalinity" is thus not understood. Further, the explanation in the description of "alkalinity" does neither clearly define the meaning of said wording (see page 3, last paragraph). Even further, in case it just means that the amount of buffering ions in the suspension is increased, this wording would be redundant since this is the implicit result of the fact that the suspension is buffered.

Claims

1. A process for stabilizing the pH of a pulp suspension in the stock preparation of a paper machine, c h a r a c t e r i z e d by increasing the alkalinity of said paper making pulp suspension by adding thereto a combination of an alkali metal hydroxide feed and a carbon dioxide feed which feeds substantially counter each other's pH changing effect, said feeds being provided in an amount sufficient to achieve a significant buffering effect of said pulp suspension while enabling utilization of an excess of said hydroxide or said carbon dioxide for adjusting the pH of said pulp suspension and maintaining the pH at a desired level throughout the paper making.

2. Process according to claim 1, c h a r a c t e r i z e d in that the pH of said pulp suspension is adjusted to a pH between about 7 and 9 by adding an excess of said alkali metal hydroxide or by adding an excess of said carbon dioxide.

3. Process according to claim 1, c h a r a c t e r i z e d in that said alkali metal hydroxide is aqueous sodium hydroxide and said carbon dioxide is gaseous carbon dioxide.

4. Process according to claim 1, c h a r a c t e r i z e d in that said alkali metal hydroxide is fed to said pulp suspension prior to the feeding of said carbon dioxide.

5. Process according to claim 1, c h a r a c t e r i z e d in that the alkalinity of said pulp suspension is increased by providing a substantially equal molar amount of alkali metal hydroxide and dissolved carbon dioxide, said amount being sufficient to provide a significant buffering effect at about pH 8.

6. Process according to claim 1, c h a r a c t e r i z e d in that said pulp suspension is chemical or mechanical pulp.

7. Process according to claim 6, c h a r a c t e r i z e d in that said pulp suspension is bleached chemical pulp.

8. Process according to claim 1, c h a r a c t e r i z e d in that said pulp suspension contains and/or is intended to contain calcium carbonate filler.

9. Process according to claim 1, c h a r a c t e r i z e d in that said alkali metal hydroxide and carbon dioxide feeds are added to said pulp suspension flowing in a pipe leading to a stock preparation tank.

10. Process according to claim 1, c h a r a c t e r i z e d in that said alkali metal hydroxide and said carbon dioxide are combined prior to feeding to the pulp suspension.

11. A process for producing paper comprising
- providing a paper making pulp suspension in the stock preparation of a paper machine;
- increasing the alkalinity of said pulp suspension by adding thereto a combination of an alkali metal hydroxide feed and a carbon dioxide feed which feeds substantially counter each others pH changing effect, said feeds being provided in an amount sufficient to achieve a substantial buffering effect of said pulp suspension while enabling utilization of an excess of said hydroxide or said carbon dioxide for adjusting the pH of said pulp supsension and for maintaining the pH at a desired level throughout the paper making;
- forming said pulp suspension into a web; and
- drying said web to form paper.

12. Process according to claim 14, c h a r a c t e r i z e d in that the pH of said pulp suspension is adjusted to a desired value between 7 and 9 by adding an excess of said alkali metal hydroxide or said carbon dioxide.

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

BORENIUS & CO. OY AB
Kansakoulukuja 3
FIN-00100 Helsinki
FINLANDE

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Rule 71.1)

Date of mailing *(day/month/year)*	12. 10. 99
Applicant's or agent's file reference 7D57PCp	**IMPORTANT NOTIFICATION**

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/FI98/00509	12/06/1998	13/06/1997

Applicant
AGA AKTIEBOLAG et al

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.

2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.

3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. **REMINDER**

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/	Authorized officer
European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Ipinazar, P Tel.+49 89 2399-8131

Form PCT/IPEA/416 (July 1992)

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
7D57PCp		
International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/FI98/00509	12/06/1998	13/06/1997
International Patent Classification (IPC) or national classification and IPC		
D21H17/64		
Applicant		
AGA AKTIEBOLAG et al		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 6 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of 2 sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☐ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   - VI ☐ Certain documents cited
   - VII ☒ Certain defects in the international application
   - VIII ☒ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
29/12/1998	12. 10. 99
Name and mailing address of the international preliminary examining authority:	Authorized officer
European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Karlsson, B Telephone No. +49 89 2399 8424

Form PCT/IPEA/409 (cover sheet) (January 1994)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/FI98/00509

I. Basis of the report

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

Description, pages:

1-10	as originally filed				

Claims, No.:

13	as originally filed				
1-12	as received on	13/08/1999	with letter of	02/08/1999	

2. The amendments have resulted in the cancellation of:

☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-12
	No:	Claims	
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1-12
Industrial applicability (IA)	Yes:	Claims	1-12
	No:	Claims	

2. Citations and explanations

see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

V

1.1 The present claim 1 defines a process for stabilizing the pH of a pulp suspension in the stock preparation of a paper machine by buffering said suspension by adding a combination of an alkali metal hydroxide and a carbon dioxide.

1.2 D3:EP-A-0 281 273, also cited in the description, discloses a process for manufacturing paper from a cellulosic pulp, which tries to improve the pH-adjustment of the alkaline pulp prior to forming paper therefrom (see D3, column 2, 1st paragraph). According to D3, gaseous carbon dioxide is added to the pulp suspension in order to adjust and maintain the pH at desired level. It is even explicitly disclosed in D3 that due to the buffering capacity of carbon dioxide, the pulp pH variations can substantially be avoided, which leads to improved properties of the resulting paper (see column 6, 1st paragraph). Moreover, it must also be considered to be a well known fact to the skilled engineer, regardless if he works in the pulp mill or paper factory, that CO_2 has a buffering capacity in aqueous solutions, i.e. in pulp suspensions (see D2: 5th Inter. conference on new available tech., The world pulp and paper week, Östberg G.M.K. (see search report), page 509, third paragraph).
Therefore, as already explained above, the skilled engineer who tries to solve the problem of adjusting the pH of pulp suspensions in the stock preparation, would surely also consider how said problem has been solved in a technically very closely related field, i.e. in the pulp mill. Thus, a skilled person would surely also consider D1:GB-A-815 247 although this document more generally deals with the manufacture of pulp without explicitly mentioning anything about the manufacture of paper therefrom. The skilled man would from this document get an indication on how the pulp suspension advantageously can be buffered, namely by using a combination of sodium hydroxide and carbon dioxide (see D1 page 2, lines 16 to 40). Thus, the combination of D3 and D1 would lead the skilled person to the subject-matter of the present claim 1.
Accordingly, the requirements of Article 33.3 PCT does not appear to be satisfied.

1.3 The features of the present claims 2,3 and 4 are already known from D1 (see above reference).
The additional features of claims 6 and 7 are known from D3 (see D3, column 1; claim 1). Further, no inventive matter appears to be present in the subject-matters of claims 5,8, 9 and 10 in the light of the disclosure of D1 and D3. Thus, presently no inventive

matter appears to be present in the dependent claims 2 to 10 in the light of the disclosures of D1 and D2 (Art. 33.3 PCT).

1.4 The wording of the present claim 11 just differs from claim 1 by the fact that it refers to a "process for producing paper" instead of a "process for stabilizing the pH of a pulp suspension in a stock preparation of a paper machine". Thus, since D3 also pertains to a process for manufacturing paper, the distinguishing features of claim 11 are then, of course, known from D3 (see D3, fig.1). Hence, by the same token as explained above, the subject-matter of claim 11 does neither satisfy the requirements of Article 33.3 PCT with regard to the disclosures of D3 and D1.

1.5 The features of the present claim 12 are known from both D3 and D1 and cannot thus be considered to add any inventive matter (see above references to D3 and D1; Art.33.3 PCT).

1.6 Although the separate features of the dependent claims not appear to add any inventive matter, the clarified claim 1, amended with a specific combination of features, might still satisfy the requirements of Articles 33.2 and 33.3 PCT.

VII

2.1 D1 and D2 should be acknowledged in the description as representing closest prior art (Rule 5.1(a)(i)-(vi) PCT).

VIII

3.1 The present claim 1 refers to a method for stabilizing the pH of a pulp suspension in the stock preparation of a paper machine. The necessary process steps to accomplish said stabilizing effect have, however, not been clearly defined in claim 1 (Art.6 PCT). It is on one hand stated that the alkalinity of the pulp suspension should be increased i.e. increasing the pH, whereas, on the other hand it is stated the pH of the pulps suspension should be adjusted by adding a excess of the hydroxide component resp. carbon dioxide, i.e. the pH can be decreased or increased. Following the examples of the present specification it becomes clear that the pH of the pulp suspension can be both decreased and increased by adding a small surplus of the

resp. buffering agents.
The exact meaning of the wording " increasing the alkalinity" is thus not understood. Further, the explanation in the description of "alkalinity" does neither clearly define the meaning of said wording (see page 3, last paragraph). Even further, in case it just means that the amount of buffering ions in the suspension is increased, this wording would be redundant since this is the implicit result of the fact that the suspension is buffered.